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Field Comparison of Polyurethane Foam and XAD-2 Resin for Air Sampling for Polynuclear Aromatic Hydrocarbons

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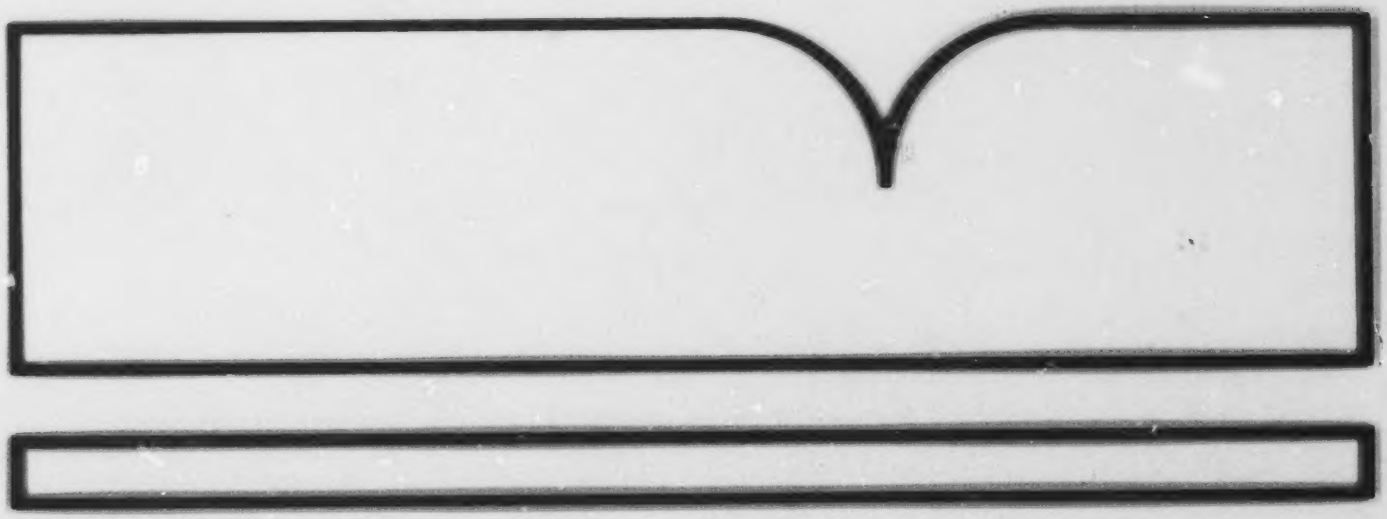

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16. ABSTRACT

A study of the sampling efficiency of two adsorbents, XAD-2 resin and polyurethane foam (PUF), for polynuclear aromatic hydrocarbons (PAH) in air, was performed under summer and winter ambient conditions. Two aspects were investigated: the collection efficiency for ambient PAH vapor and the retention efficiency for PAH spiked onto the adsorbents before sampling. XAD-2 resin had a higher collection efficiency for 2ring PAH and a higher retention efficiency for volatile or reactive PAH than did PUF. The recoveries of volatile PAH using PUF were improved at lower temperatures, but the recoveries of reactive PAH, such as cyclopenta[c,d]pyrene (CcdP), were not improved at lower temperatures using either adsorbent. The stability of PAH collected on quartz fiber prefilters and XAD-2 or PUF back-up traps as a function of the storage time was also studied. Storage at room temperature in the dark for 30 days did not affect the levels of PAH collected or spiked on XAD-2 resin. However, the levels of naphthalene, anthracene, and benzo[a]pyrene decreased during storage. With one exception, CcdP, the levels of particle-bound PAH collected on filters were not affected significantly during storage. The distributions of PAH between filters and adsorbent agreed with the volatilities of the PAH, and most 2- to 4-ring PAH broke through the filters. This demonstrated that the use of both prefilters and back-up adsorbents is necessary in air sampling for PAH.

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Field Comparison of Polyurethane Foam and XAD-2 Resin for Air Sampling for Polynuclear Aromatic Hydrocarbons

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■ A study of the sampling efficiency for polynuclear aromatic hydrocarbons (PAH) in air of two adsorbents, XAD-2 resin and polyurethane foam (PUF), was performed under summer and winter ambient conditions. Two aspects were investigated: (1) collection efficiency for ambient PAH vapor and (2) retention efficiency for native and perdeuteriated PAH spiked onto the adsorbents before sampling. The XAD-2 resin had a higher collection efficiency for naphthalene than did PUF. Some spiked PAH that were volatile or reactive were also recovered more efficiently with XAD-2 resin than with PUF. Lower sampling temperatures improved the recoveries of volatile PAH with PUF, but the recoveries of reactive PAH, such as cyclopenta[*cd*]pyrene, were not improved at lower temperatures for either adsorbent. The stability of PAH collected on quartz-fiber prefilters and XAD-2 or PUF backup traps as a function of the storage time was also investigated. Storage at room temperature in the dark for 30 days did not have an adverse effect on ambient PAH collected with XAD-2 resin or on perdeuteriated PAH spiked onto the adsorbent prior to sampling. However, a decreasing concentration trend with storage time for naphthalene, anthracene, and [$^2H_{12}$]benzo[*a*]pyrene collected or spiked on PUF was found. Most of the particle-bound PAH collected on quartz-fiber filters were not affected significantly during storage, except that cyclopenta[*cd*]pyrene decreased to approximately half the original level after 30 days of storage.

Introduction

Polynuclear aromatic hydrocarbons (PAH) have been studied extensively and have received increased attention in studies of air pollution in recent years because some of these compounds are potent carcinogens, mutagens, or both (*1–5*). To understand the extent of human exposure to PAH, reliable sampling and analytical methodology must be established for monitoring the concentrations of PAH in air. In general, the analytical methodology is well developed; however, the ambiguities introduced by sampling procedures can often reduce the validity of conclusions based on the analytical results. Several studies (*6–9*) have shown that PAH having two to four rings are present in air primarily in the vapor phase and are not retained by filters because of their volatility.

With regard to health effects, the collection of gas-phase PAH in air may be as important as the collection of particle-bound PAH. Thus, a wide variety of adsorbents such as XAD-2 resin, Tenax GC, and polyurethane foam (PUF) has been used to collect PAH vapors (*10–14*). All these adsorbents have been demonstrated to have a high collection efficiency for certain PAH. Typically, Tenax GC is used for sampling volatile analytes for subsequent thermal desorption rather than for semivolatile analytes for subsequent solvent elution, for which PUF and XAD-2 are used. Therefore, Tenax GC was excluded from this study. Polyurethane foam has relatively better flow characteristics than XAD-2 resin and is easy to handle in the field. However, PUF was found to give rise to unknown components that interfered with the PAH analyses, even after extensive cleanup by Soxhlet extraction (*14*). A new cleanup procedure was developed (*12*) that was found to eliminate the interferences. This new procedure was employed in this study. The overall comparative effectiveness of these adsorbents and the degree to which quantification of PAH is affected by sampling, handling, and storage have not been fully investigated. This uncertainty precludes an accurate assessment at this time of

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the relative overall merit of each adsorbent.

The investigation reported here had two goals: (1) comparison of two adsorbents, PUF and XAD-2 resin, for their effectiveness in air sampling of PAH vapors and (2) comparison of the stability of PAH vapors collected on PUF and XAD-2 resin and of particle-bound PAH collected on quartz-fiber filters, as a function of the storage time between collection and extraction.

Experimental Section

Adsorbent Preparation. The PUF cartridges were purchased from Southwest Research Institute. The modified and improved cleanup procedure included sequential (50 times) compression/decompression of the PUF plug in 800 mL each of toluene, acetone, and 10% ether/hexane. The PUF plug was then Soxhlet extracted with acetone for 16 h and dried in a vacuum oven for 6 h at room temperature. The PUF plug was placed in a clean glass cartridge (2.3-in. i.d., 5-in. length), wrapped with hexane-rinsed aluminum foil, and stored in a clean screw-capped jar. All PUF plugs were cleaned by this procedure 24 h before air sampling.

The XAD-2 resin, which is a styrene–divinylbenzene polymer, was purchased as a precleaned resin from Supelco and was further cleaned by Soxhlet extraction with methylene chloride for 16 h and dried with a nitrogen gas stream. The clean XAD-2 resin was placed in the same type of glass cartridge described above. A nickel wire screen was placed in the bottom of the glass cartridge to retain the resin. The nickel wire screen was purchased from UNIQUE Wire Weaving Co. (mesh size 200/200, wire size 0.0021–0.0022 in.). The XAD-2 resin bed depth was 2 in., which allowed a maximum sampling flow rate of approximately 7 cfm.

Sampling Apparatus and Location. Modified medium-volume samplers (modified General Metals PS-1 samplers with General Metals bypass motors) were employed in the ambient air sampling. The samplers, which are described elsewhere (*15*), were equipped with a shelter that shields the filter and the PUF or XAD-2 backup trap from sunlight during sampling. Samplers were placed on the ground in an open space outside Battelle's laboratories in Columbus, OH. This location can be classified as a medium size, midwestern city with relatively clean air quality for an urban environment. The sampling was performed on weekends or holidays to reduce the contribution of local vehicle exhaust emissions to the sample. A 5-ft exhaust hose leading away from the sample module was attached to each sampler. Each sampler was calibrated with a dry gas meter (Rockwell Model 415) to obtain a flow rate of 6.7 cfm. Flow measurements from the sampler Magnehelic gauge were then recorded. The sample module was then placed on the sampler, and adjustment was made by the orifice calibrator to obtain the same calibrated reading on the Magnehelic gauge. Air was sampled for 24 h at 6.7 cfm. The flow was checked approximately every 6 h and maintained at 6.7 cfm throughout the sampling period. The same flow was used for both PUF and XAD-2. To ensure that only vapor-phase PAH were collected on the PUF and XAD-2, quartz-fiber filters (104-mm QAST, Pallflex) were located upstream of the PUF or XAD-2 in all sampling experiments (both the sampling efficiency comparision and the storage stability studies).

Sampling Procedure. (A) PUF and XAD-2 Sampling Efficiency Comparison. In the PUF and XAD-2 sampling efficiency comparison, two sets of three samplers were located in parallel, approximately 2 ft apart. The three samplers of each set were separated from each other by approximately 1 ft. In one set of three samplers PUF cartridges were used, and in the other set XAD-2 cartridges were used. Just prior to sampling, two each of the three PUF and XAD-2 cartridges were spiked with 100 μL of a methylene chloride solution containing 2.0 μg of each of the target PAH. The target PAH were [$^2H_8$]naphthalene, [$^2H_{10}$]phenanthrene, [$^2H_{10}$]pyrene, [$^2H_{12}$]chrysene, [$^2H_{12}$]benzo[*a*]pyrene, phenanthrene, anthracene, fluoranthene, pyrene, cyclopenta[*cd*]pyrene, benz[*a*]anthracene, benzo[*e*]pyrene, and benzo[*a*]pyrene. The spiking was performed by injecting the solution into the center of the PUF plug or XAD-2 resin bed at about 1-in. depth, followed by 5 min of air-drying. These experiments were performed twice under the same conditions except that during the first experiments the ambient temperature ranged from 66 to 86 °F and during the second experiments it ranged from 17 to 27 °F.

(B) Sampling Module Storage Stability Study. In the storage stability study, eight samplers in two sets of four were used. Sampler location was similar to the comparison study described above. In the first experiment, PUF cartridges were used for all eight samplers, and in the second experiment, XAD-2 cartridges were used for all samplers. All PUF and XAD-2 cartridges were spiked before sampling as described above but with only the perdeuteriated PAH. After the sampling, the PUF and XAD-2 cartridges were stored in the dark at room temperature for 0-, 10-, 20-, and 30-day intervals before extraction and were analyzed as replicate pairs. The eight quartz-fiber filters obtained from the XAD-2 sampling were similarly stored and extracted after the same time intervals. The filters from the PUF sampling were not used in this study.

Analytical Method. Quartz-fiber filters, PUF, and XAD-2 samples were Soxhlet-extracted for 16 h with 10% ether/hexane (PUF) or methylene chloride (filters and XAD-2). The extracts were concentrated to 1 mL with Kuderna–Danish (K–D) evaporation and analyzed by 70-eV electron impact (EI) gas chromatography/mass spectrometry (GC/MS). A Finnigan 4500 quadrupole GC/MS system and an INCOS 2300 data system were used. The evaporator cavity GC injector at 300 °C was used in the splitless mode (55 s) for a 2.0-μL injection. The GC column was an Ultra No. 2 fused silica capillary column (50-m length, 0.31-mm i.d., 0.17 μm film thickness, Hewlett-Packard Co.), and the column outlet was located in the MS ion source. Following injection, the GC was held at 45 °C for 1.5 min and temperature programmed to 100 °C over 5 min and then to 320 °C at 6 deg/min. The molecular ion of each compound of interest was monitored in the multiple ion detection mode (*16*). Identifications of PAH were based on the GC retention times of the individual monitored molecular ion signals relative to that of the internal standard 9-phenylanthracene. Quantifications of PAH were based on comparisons of the respective integrated ion current responses for the monitored molecular ions to that of the internal standard, with calibration response curves generated from four different concentrations (0.1 ng/μL, 2.0 ng/L, and 5.0 ng/L) of each target PAH.

Results and Discussion

PUF and XAD-2 Sampling Efficiency Comparison. PAH recoveries from the PUF and XAD-2 resin were determined by subtracting the levels of PAH in a nonspiked background sample from those in the corresponding spiked samples and dividing by the spiked levels (2.0 μg). The PAH recovery data are summarized in Figures 1 and

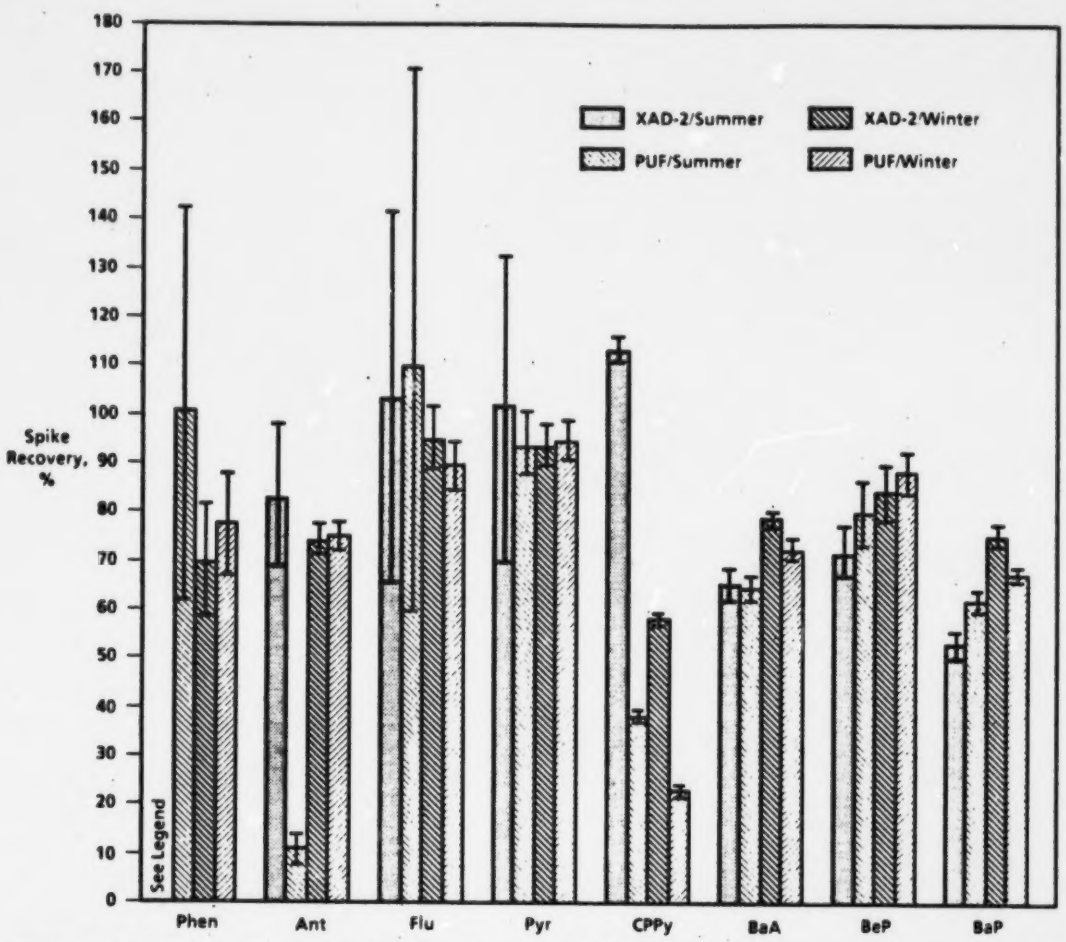

Figure 1. Recovery of spiked native PAH from XAD-2 resin and PUF. Vertical bars represent the average recoveries from the duplicate pairs, shown as the range of deviation from the average. Phe, phenanthrene; Ant, anthracene; Flu, fluoranthene; Pyr, pyrene; CPPy, cyclopenta[*cd*]pyrene; BaA, benz[*a*]anthracene; BeP, benzo[*e*]pyrene; BaP, benzo[*a*]pyrene. Phenanthrene recovery data in the summer experiment cannot accurately be calculated because of high background levels.

Table I. Background Concentrations of PAH in Air Collected on PUF and XAD-2 Resin[a]

	summer experiment			winter experiment		
	concn, ng/m³		ratio,	concn, ng/m³		ratio,
compound	XAD-2	PUF	XAD-2/PUF	XAD-2	PUF	XAD-2/PUF
naphthalene	182	7.7	24	68	5.5	12
phenanthrene	152	134	1.1	17	17	1.0
anthracene	3.0	3.8	0.8	0.8	0.9	0.9
fluoranthene	37	47	0.8	2.6	3.4	0.8
pyrene	11	13	0.8	1.7	2.1	0.8
cyclopenta[*cd*]pyrene	ND[b]	ND		ND	ND	
benz[*a*]anthracene	ND	ND		ND	ND	
benzo[*e*]pyrene	ND	ND		ND	ND	
benzo[*a*]pyrene	ND	ND		ND	ND	

[a] A total of 273 m³ of air was sampled with a given sampler in a given run in both summer and winter experiments. [b] ND = not detected.

2. The background PAH levels collected on the PUF and XAD-2 resin are given in Table I. On the basis of our previous studies with these sampling and analysis procedures, a variance of approximately 20% for identical samples is to be expected. Thus, the PUF and XAD-2 results in Table I are similar for all compounds except naphthalene, the most volatile PAH of the series. As shown in Table I, levels of ambient naphthalene adsorbed on XAD-2 resin are about 24 and 12 times higher than those on PUF in the summer and winter experiments, respectively. The summer phenanthrene levels are slightly higher for XAD-2 resin than for PUF, but identical levels were observed for both adsorbents in the winter experiment. This finding suggests that XAD-2 resin has better collection efficiency for volatile PAH than does PUF and that the collection efficiency for volatile PAH on PUF improves at lower ambient sampling temperatures. In addition, only two- to four-ring PAH are found in both adsorbents. As we expected, the nonvolatile, high molecular weight PAH were predominantly present as ad-

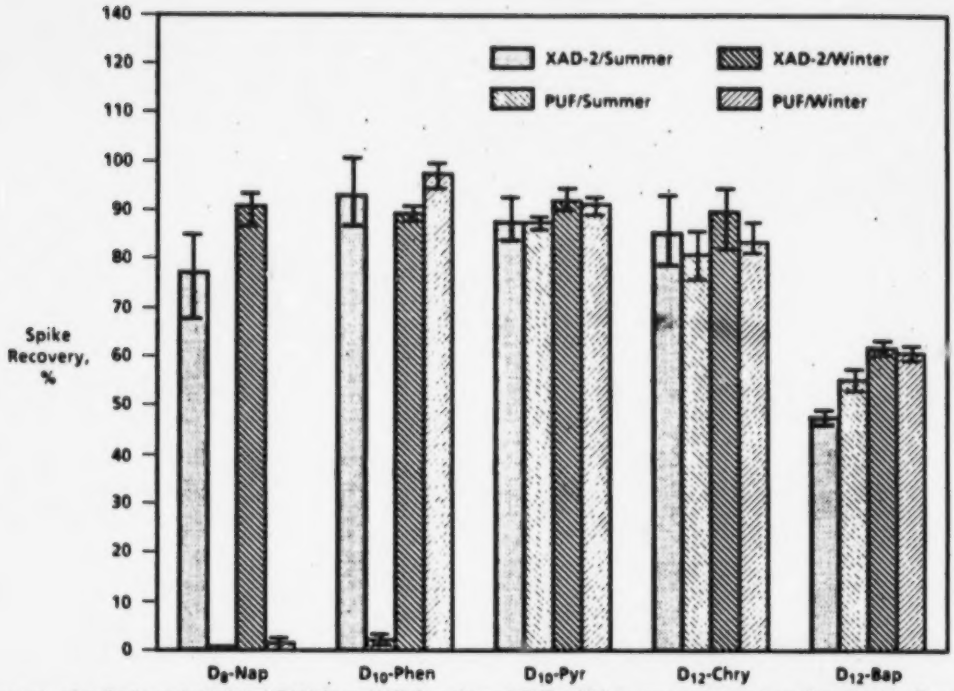

Figure 2. Recovery of spiked perdeuteriated PAH from XAD-2 resin and PUF. Values are expressed as described in Figure 1. D_8-Nap, [$^2H_8$]naphthalene; D_{10}-Pyr, [$^2H_{10}$]pyrene; D_{12}-Chry, [$^2H_{12}$]chrysene; D_{12}-BaP, D_{12} [$^2H_{12}$]benzo[*a*]pyrene.

sorbed species on particulate matter and were thus retained on the filter.

As shown in Figures 1 and 2, generally good recoveries are obtained for most spiked PAH from both adsorbents after exposure to ambient air at a flow rate of 6.7 cfm for 24 h. Comparison of the [$^2H_8$]naphthalene recoveries shows that the spiked XAD-2 resin give substantially higher recoveries than the spiked PUF, in agreement with the finding for native naphthalene noted above. Thus, XAD-2 resin appears to be significantly more effective in retaining two-ring PAH, since approximately 80% of [$^2H_8$]naphthalene was recovered after ambient air exposure even at the summer sampling temperature (66–86 °F). Similarly, lower recoveries of the spiked [$^2H_{10}$]-phenanthrene and spiked native anthracene were obtained from PUF than from XAD-2 resin in the summer experiment. As expected, these PUF recoveries improved significantly in the winter experiment, becoming comparable to those from XAD-2 resin. In an apparent contradiction to this observation with spiked deuteriated compounds, the background levels of ambient phenanthrene and anthracene (Table I) did not differ significantly between XAD-2 and PUF. The discrepancy is explainable in terms of more facile breakthrough of the spiked PAH than of ambient native PAH vapor. The spike was added as a liquid to the center of the adsorbent at approximately 1-in. depth and, therefore, had achieved 20% of its breakthrough for PUF and 50% breakthrough for XAD-2 before sampling began. In addition, the concentrated application of the spiked compounds might have created a localized adsorbent overload that could have increased the breakthrough tendency of these spike PAH. Nevertheless, the results clearly show that temperature is an important factor to be considered in ambient air sampling.

It was noted that the phenanthrene recovery data cannot be accurately addressed in the summer experiment because the ambient air background levels were about 19 times higher than the spiked levels. Thus, due to the expected variations in sampling and analytical procedures, negative recoveries were sometimes observed for spiked phenanthrene after subtraction of these high ambient background levels. For the same reason, a greater variance for fluoranthene recoveries was observed from both adsorbents in the summer experiment. In the winter experiment, the phenanthrene and fluoranthene background levels were only about 2.5 and 0.5 times the spiked levels, respectively, and good recoveries were obtained from both adsorbents. Low recoveries of cyclopenta[*cd*]pyrene were obtained with PUF in both the summer and the winter experiments, 40 and 23%, respectively. The recoveries of this compound from spiked XAD-2 resin also showed a similar trend, decreasing from 100% (summer) to 58% (winter). The loss of spiked cyclopenta[*cd*]pyrene almost certainly results not from volatilization but from decomposition, possibly oxidation; this is also indicated by the storage stability results (see below). The recoveries of the remaining PAH were good, and better retention of some PAH was observed at lower sampling temperatures for both adsorbents.

Sampling Module Storage Stability Study. The stability study was conducted to determine whether a significant loss of PAH adsorbed on PUF, XAD-2, or quartz-fiber prefilters occurs during the storage period between collection and extraction. Since the experimental design required duplicate samples for four storage times and since only eight medium-volume samplers were available, only perdeuteriated PAH were spiked onto the adsorbents prior to sampling. Thus, comparisons of the native PAH levels can be made without any background level corrections. The results are summarized in Figure 3 and 4.

For XAD-2 resin, no loss of perdeuteriated PAH was detected over the 30-day storage interval. For PUF, the [$^2H_8$]phenanthrene storage stability results are not meaningful since only 1.2% of the spiked [$^2H_8$]naphthalene was retained on the PUF at the end of the 24-h sampling. The decrease in [$^2H_{12}$]benzo[*a*]pyrene, however, does apparently indicate storage instability of this compound on PUF, since about 50% recovery of this spiked analyte was

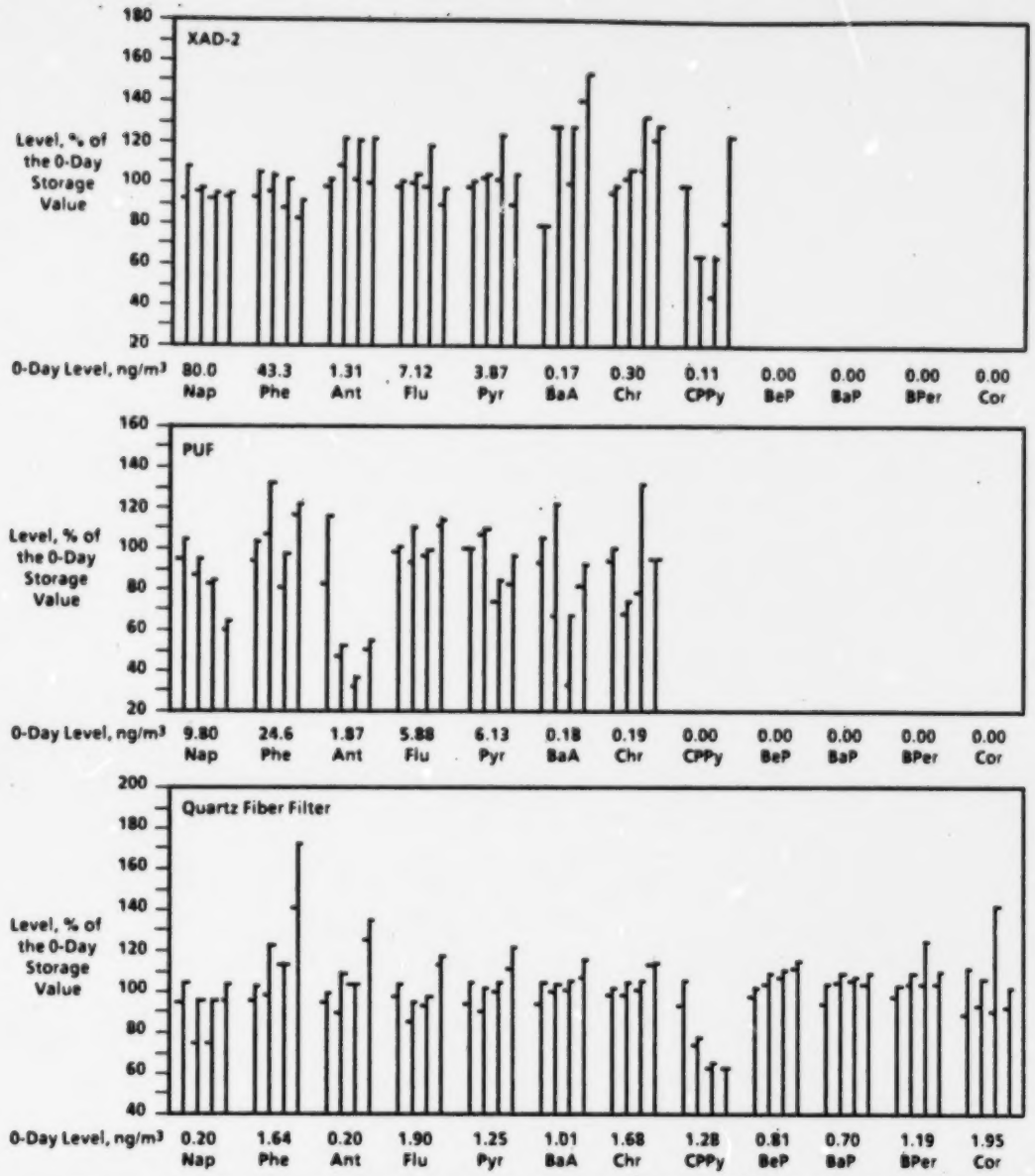
XAD-2
Level, % of the 0-Day Storage Value
0-Day Level, ng/m³
80.0 43.3 1.31 7.12 3.87 0.17 0.30 0.11 0.00 0.00 0.00 0.00
Nap Phe Ant Flu Pyr BaA Chr CPPy BeP BaP BPer Cor
PUF
Level, % of the 0-Day Storage Value
0-Day Level, ng/m³
9.80 24.6 1.87 5.88 6.13 0.18 0.19 0.00 0.00 0.00 0.00 0.00
Nap Phe Ant Flu Pyr BaA Chr CPPy BeP BaP BPer Cor
Quartz Fiber Filter
Level, % of the 0-Day Storage Value
0-Day Level, ng/m³
0.20 1.64 0.20 1.90 1.25 1.01 1.68 1.28 0.81 0.70 1.19 1.95
Nap Phe Ant Flu Pyr BaA Chr CPPy BeP BaP BPer Cor

Figure 3. Levels of native PAH recoveries with XAD-2 resin, PUF, and quartz-fiber filter as a function of storage time. Chr, chrysene; BPer, benzo[*ghi*]perylene; Cor, Coronene. The remaining compound names are expressed as described in Figure 1. The four vertical bars for each PAH representing 0-, 10-, 20-, and 30-day storage intervals are shown from left to right, and values for each of two replicates, expressed as percent of the average 0-day storage values, are shown as horizontal bars.

also found for the sampling efficiency study (see Figure 2). However, this may not cause a serious problem in ambient air sampling, because benzo[*a*]pyrene is usually retained on the particulate filter and is not present in the backup adsorbent.

As shown in Figure 3, storage instability over the 30-day period is not indicated for any of the native PAH collected on XAD-2 resin. Only trace amounts of cyclopenta[*cd*]-pyrene were found on XAD-2 resin, and the variability in the data at these low levels prevents any firm stability conclusion. However, the data are consistent with a decreasing trend with storage time.

For PUF adsorbent, there appears to be a loss on storage of both native naphthalene and native anthracene. Since naphthalene evidently breaks through PUF during a 24-h sampling, it is not surprising that it could be partially lost by volatilization after storage for 30 days. Unfortunately, this hypothesis cannot be supported by the [$^2H_8$]-naphthalene PUF results because only 1.2% of the spiked material was present at the beginning of storage and the recovery levels are too low to show any trend. The greater loss from PUF of anthracene relative to its isomer phenanthrene is more consistent with the lower chemical stability of anthracene relative to phenanthrene than with a selective loss through volatilization. The nondetection of cyclopenta[*cd*]pyrene on PUF may be due to its degradation to below the detection limit (0.05 ng/m^3) since this compound was detected only at very low levels with XAD-2 resin. An overall comparison of the XAD-2 vs. PUF results of Figure 4 clearly suggests more reliable and reproducible storage performance for XAD-2 resin.

With the exception of cyclopenta[*cd*]pyrene, storage losses of particle-bound PAH collected on quartz-fiber filters were insignificant. The calculated concentration of

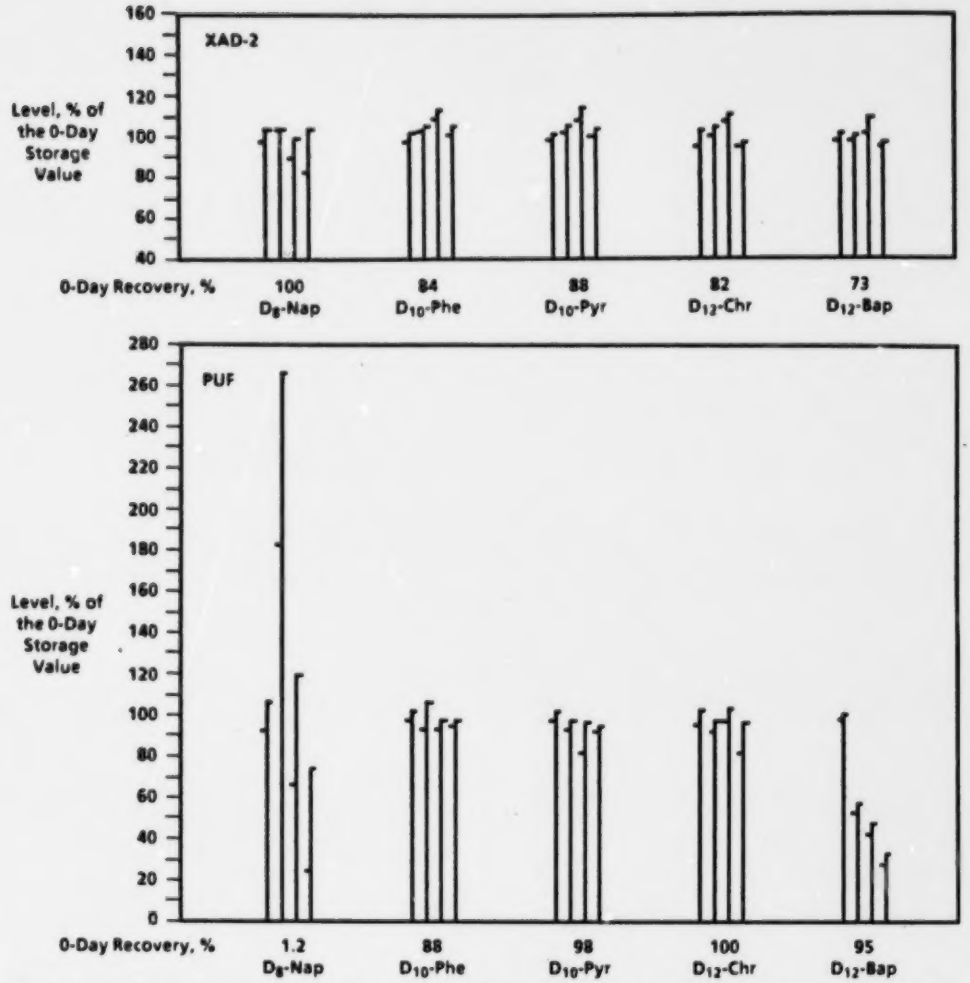

Figure 4. Recovery of spiked perdeuteriated PAH in XAD-2 resin and PUF as a function of storage time. Compound names are expressed as described in Figure 2. Values are expressed as described in Figure 3.

cyclopenta[*cd*]pyrene decreased from 1.3 ng/m^3 (34.8 ng/mg of particles) to 0.8 ng/m^3 (17.8 ng/mg of particles) after storage for 30 days. The losses of particle-bound cyclopenta[*cd*]pyrene during sample storage are probably due to its chemical instability noted above in the discussion of its low recovery in the sampling efficiency study (Figure 1). One research group (*17*) has identified a direct-acting mutagen, pyrene-1,10-dicarboxylic acid anhydride, as a possible degradation product of cyclopenta[*cd*]pyrene in diesel exhaust particles. This acid anhydride oxidation product was also found in airborne particulate material and other environmental samples by other research groups (*18–19*).

Distributions of PAH between the filter and XAD-2 resin from the stability study were as follows (Figure 3): two-ring PAH, 99% on XAD-2; three-ring PAH, 90% on XAD-2; four-ring PAH, 80% on XAD-2 for pyrene and fluoranthene and 15% on XAD-2 for benz[*a*]anthracene and chrysene; five-ring PAH, <10% on XAD-2; six- to seven-ring PAH, 100% on filter. These distributions parallel, in general, the volatilities of the individual PAH and indicate breakthrough of two- to four-ring PAH from the prefilters. Other research groups (*6–9*) have also reported that most of the volatile PAH were not retained on filters under high-volume sampling conditions. In our study, medium-volume samplers were used, and the flow rate (6.7 cfm) was lower than a typical high-volume sampling flow rate. These relative recovery results clearly demonstrate that the use of filters only to collect PAH from air is not sufficient, and correct quantitative results, even for species as large as chrysene, cannot be obtained without the use of backup adsorbents.

Conclusions

In conclusion, for air sampling for gas-phase PAH, XAD-2 resin seems to gave two performance advantages compared to PUF: (1) two- and three-ring PAH clearly break through standard PUF cartridges with a typical sampling volume of 270 m^3, and (2) PAH species that are prone to degradation during sampling or during sample storage prior to extraction exhibit greater losses from PUF than from XAD-2 resin. The retention efficiency of volatile PAH for both adsorbents, especially PUF, can be improved by lowering the sampling temperature. Certainly, the future use of quartz-fiber filter/PUF samplers should involve a minimum of sample handling and storage to reduce the losses of volatile and reactive PAH. Little is known about the degradation products of PAH due to sampling and sample handling. The critical issue of whether a significant part of the mutagenic activity in these types of air samples is from sampling artifacts or is actually present in the sampled air is still not clear, and further

investigations need to be carried out in this area.

Acknowledgments

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Registry No. XAD-2, 9060-05-3; Chr, 218-01-9; BPer, 191-24-2; Cor, 191-07-1; naphthalene, 91-20-3; phenanthrene, 85-01-8; anthracene, 120-12-7; fluoranthene, 206-44-0; pyrene, 129-00-0; cyclopenta[*cd*]pyrene, 27208-37-3; benz[*a*]anthracene, 56-55-3; benzo[*e*]pyrene, 192-97-2; benzo[*a*]pyrene, 50-32-8.

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